PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Zaki *et al.* Group Art Unit: 1796

Serial No.: 10/791,427 Examiner: Del Cotto, Gregory R.

Filed: March 2, 2004 Docket No.: 1456/3

Confirmation No.: 3775

For: SOLVENT COMPOSITIONS FOR REMOVING PETROLEUM RESIDUE FROM A SUBSTRATE AND METHODS OF USE THEREOF

* *

SECOND DECLARATION OF DR. ROBERT E. TROXLER
PURSUANT TO 37 C.F.R. §1.132

Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Sir:

1. My name is Dr. Robert E. Troxler, and I am the Director of Advanced Technologies at Troxler Electronic Laboratories, Inc. of Research Triangle Park, North Carolina, assignee for the subject U.S. Patent Application Serial No. 10/791,427. I am also a co-inventor of the subject U.S. Patent Application Serial No. 10/791,427.

2. A true and accurate copy of my *curriculum vitae*, which evidences my expertise and credentials, is being submitted herewith as **Exhibit A**.

3. I have had an opportunity to review pending claims 1-42 and 105-135 in the above captioned U.S. Patent Application Serial No. 10/791,427.

4. I have also reviewed the following documents: the Non-Final Official Action dated July 28, 2010 on the above captioned U.S. Patent Application Serial No. 10/791,427 (hereinafter the "Official Action") by the U.S. Patent and Trademark Office

(hereinafter "the Patent Office"); U.S. Patent No. 6,281,189 to Heimann *et al.* (hereinafter "Heimann"); U.S. Patent Application Publication No. US 2003/0213747 of Carbonell *et al.* (hereinafter "Carbonell"); U.S. Patent No. 5,143,639 to Krawack (hereinafter "Krawack"); U.S. Patent No. 5,413,729 to Gaul (hereinafter "Gaul"); U.S. Patent No. 5,421,907 to Nieendick *et al.* (hereinafter "Nieendick"); U.S. Patent No. 5,194,173 to Folkard *et al.* (hereinafter "Folkard"); U.S. Patent No. 6,030,466 to Myers, II (hereinafter "Myers, II"); U.S. Patent No. 6,544,942 to Smith *et al.* (hereinafter "Smith"); U.S. Patent No. 6,838,426 to Zeilinger (hereinafter "Zeilinger"); and U.S. Patent No. 7,547,672 to Zaki (hereinafter "Zaki").

5. I have performed and/or supervised several experiments designed to test the abilities of various compositions to dissolve asphalt. The tests employed were based on the North Carolina Department of Transportation testing regimen disclosed in Whitley (2003) "Developing an Asphalt Solvent Testing and Approval Program in North Carolina", *Transportation Research Circular* (Transportation Research Board of the National Academies, Washington, D.C.). No. E-C052 pgs. 133-141, a true and accurate copy of which is being provided herewith as **Exhibit B**. The compositions tested included diesel fuel, biodiesel, methyl soyate, and exemplary solvents disclosed in Krawack and Heimann.

6. With respect to the exemplary solvents disclosed in Krawack and Heimann that were tested, these solvents had the following compositions:

- U.S. Patent No. 5,143,639 (Krawack): 25% canola oil; 25% vegetable oil; 40% water; and 10% CO-40 (surfactant)
- U.S. Patent No. 6,281,189 (Heimann): 40% methyl soyate; 40% D-limonene; and 20% water

7. For comparison, an exemplary solvent of the subject matter disclosed and claimed in the above captioned U.S. Patent Application Serial No. 10/791,427 ("BindOff"), was also tested. The particular BindOff employed in the tests had the

following composition: 59.95% IPB by volume; 39.94% biodiesel by volume; and 0.1% fragrance. The results of these tests are provided in Table 1.

Table 1

Performance Comparison of Various Solvents

Solvent Used	Avg. Mass Bitumen Removed/Mass Solvent (g/g)	Avg. Corrected Bitumen/Solvent Ratio (g/g%)
Diesel Fuel	5.8	16
Biodiesel	-7.8	2.4
Methyl Soyate	-10	0.2
Heimann	-5	5.2
Krawack	-1	9.2
BindOff	13	23.2

Correction Factor: 10.2. This correction is based on the industry standard that sets the efficiency of diesel fuel at 16%. Given that diesel fuel was determined to be 5.8 in this measure, 10.2 has been added to each measurement to thus "correct" diesel fuel to the accepted 16%. It is my understanding that this correction factor is common in the asphalt industry.

8. As shown in Table 1, BindOff was substantially more efficient at dissolving and removing bitumen than either diesel fuel or any of the aliphatic ester solvents, including those that were exemplary solvents disclosed in Krawack or Heimann. In related experiments (not all data is being shown) and depending on the exact composition of the solvent of the subject matter of the above captioned U.S. Patent Application Serial No. 10/791,427 that was tested, the various solvents of the subject matter of the above captioned U.S. Patent Application Serial No. 10/791,427 have been observed to remove 21-26% of the bitumen present in testing. By comparison, the next best solvent, diesel fuel, typically removed about 16% or less. The other solvents tested were an exemplary solvent disclosed in Krawack, an exemplary solvent disclosed in Heimann, Biodiesel (B-100), and Methyl Soyate. Methyl Soyate and Biodiesel are commercially available, environmentally friendly solvents that are commonly used in

industry. As the representative data presented in Table 1, neither performed well. The exemplary solvents based on the disclosures of Krawack and Heimann were also poor.

9. In further testing, different mixtures of aliphatic and aromatic esters were tested to determine the abilities of these mixtures to dissolve asphalt for comparison to aliphatic and aromatic esters alone. These experiments employed biodiesel as an exemplary aliphatic ester solution and isopropyl benzoate (IPB) as an exemplary aromatic ester. Eleven (11) different compositions were produced and tested in duplicate in a manner similar to the North Carolina Department of Transportation testing regimen referenced above but with actual aggregate used as the substrate, with each composition containing from 0-100% biodiesel in 10% increments, with the remainder (if any) being IPB (*i.e.*, 100-0% IPB) in 10% increments. The results are summarized in Tables 2 and 3:

Table 2

Asphalt Removal of Biodiesel/IPB Mixtures

Biodiesel Vol %	IPB Vol %	Average Initial Mass Asphalt (g)	Average Final Mass Asphalt (g)	Average Fraction Removed
0	100	2.97	2.63	0.1150
10	90	1.78	1.53	0.1432
20	80	1.81	1.55	0.1443
30	70	2.46	2.15	0.1280
40	60	2.27	2.00	0.1173
50	50	1.56	1.34	0.1347

60	40	1.90	1.74	0.0858
70	30	1.99	1.82	0.0896
80	20	2.90	2.72	0.0638
90	10	2.67	2.49	0.0670
100	0	2.41	2.29	0.0487

Table 3

Performance Comparisons of Various Mixtures

Biodiesel:IPB	Fold Improvement over 100% Biodiesel	Fold Improvement over 100% IPB	Expected Average Fraction Removed	Percent of Expected
0:100	2.3596	1.0000	0.1150	100
10:90	2.9392	1.2456	0.1084	132.14
20:80	2.9610	1.2548	0.1017	141.89
30:70	2.6270	1.1133	0.0951	134.63
40:60	2.4059	1.0196	0.0885	132.49
50:50	2.7643	1.1715	0.0819	164.49
60:40	1.7600	0.7459	0.0753	113.91
70:30	1.8392	0.7795	0.0686	130.67
80:20	1.3097	0.5550	0.0620	102.95
90:10	1.3739	0.5823	0.0553	121.08
100:0	1.0000	0.4238	0.0487	100

10. In Table 2, the "Average Fraction Removed" is expressed as the average of the two tests calculated as the initial mass of asphalt minus the final mass of asphalt, with the result divided by the initial mass of asphalt. In Table 3, the "Expected Average Fraction Removed" was calculated as the sum of the Biodiesel percentage (divided by 100) present in the mixture x 0.0487 (*i.e.*, the performance of 100% Biodiesel) + the IPB percentage (divided by 100) present in the mixture x 0.1150 (*i.e.*, the performance of 100% IPB). For example, for the 50:50 mixture, the expected average concentration removed was (0.5 x 0.0487) + (0.5 x 0.1150), which equals 0.0819. Since this mixture actually removed 1.1715, the "Percent of Expected" is [0.1347/0.0819] x 100, or 164.49%, which means that the 50:50 mix performed 64.49% better than expected.

11. Reviewing the data presented in Tables 1-3, it is believed that the compositions disclosed and claimed in the above captioned U.S. Patent Application Serial No. 10/791,427 provide a level of solvency that is unexpectedly superior to what might have been predicted based on the individual solvencies of the components of the mixtures.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Dr. Robert Troxler

Dr. Robert Troxler

September 14, 2011

Date

Attachments: **Exhibits A** and **B**

Exhibit A

Robert Ernest Troxler Ph.D.

Raleigh, North Carolina 27605

Interests:

Industrial research and development. To take part in the fundamental research and design of electromagnetic components, systems or commercial products. My interests include: radar systems, antennas, telecommunications, superconducting devices, and non-destructive evaluation of materials. Over 20 years of extensive experience in measuring the dielectric properties of materials from DC to the mm band.

Education:

Doctor of Philosophy in Electrical Engineering, Georgia Institute of Technology, December, 1992.

Master of Science in Electrical Engineering, Georgia Institute of Technology, June, 1986.

Bachelor of Science in Electrical Engineering, North Carolina State University, December 1983.

Honors: NASA GSRP Fellow, EMS Fellow, Eta Kappa Nu, Tau Beta Pi.

Experience:

February 2011: Strategic Advisory Board of the Department of Electrical Engineering, North Carolina State University

2010-present: National Center for Asphalt Technology (NCAT) Applications Steering Committee member, National Center for Asphalt Technology, Auburn University, Auburn Alabama.

April 1998-2009: Director of the North Carolina State University Engineering Foundation

April 1997: Director of Advanced Technologies, Troxler Electronic Laboratories Inc., Research Triangle Park, North Carolina

January 1994-present: Research Department, Troxler Electronics Laboratories Inc.

Exhibit A

December 1992: Doctor of Philosophy in Electrical Engineering, Georgia Institute of Technology. Dissertation Title: Magnetic Control of Superconducting Phase-Shifters with Experimental Verification.

June 1992 to December 1992: Awarded a fellowship sponsored by Electromagnetic Sciences Incorporated, Atlanta Georgia.

June 1989-June 1992: NASA Fellow (Graduate Student Researchers Program), Marshall Space Flight Center, Huntsville, Alabama, Georgia Institute of Technology, Atlanta Georgia. Research Statement: The objective of this research was to investigate the feasibility of controlling the phase velocity of thin film superconducting delay lines with a biasing magnetic field or transport current.

November 1990-November 1991: On site at Marshall Space Flight Center (Space Science Laboratory) to perform the experimental part of my thesis. Participated in a flight experiment to study the effects of atomic oxygen on many space materials. Deposited many thin films for this experiment.

September 1984-June 1989: Graduate Research (Teaching) Assistant Georgia Institute of Technology, Atlanta, Georgia. Taught Graduate Microwave Laboratory utilizing a HP-8510 Automatic Network Analyzer. Researched procedures of cryogenic microwave measurements. Researched possible microwave applications of High Temperature Superconductors supported by internal "seed funds". Wrote a laboratory procedures manual consisting of passive microwave devices for the Graduate Microwave Laboratory. Researched the possibility of using ferrite loaded image lines as phase-shifters.

December 1983-September 1984: Design Engineer, Troxler Electronic Laboratories Inc. Designed microprocessor based portable equipment for nondestructive evaluation of materials incorporating nuclear isotopes.

U.S. Patents Issued:

6,579,500: Apparatus and method for determining weight loss of a heated material

6,567,498: Low activity nuclear density gauge

6,492,641: Apparatus and method for gamma-ray determination of bulk density of samples

Exhibit A

6,442,232: Thin layer nuclear density gauge

6,440,746: Method and apparatus for analyzing asphalt content

6,436,718: Apparatus and method for determining weight loss of a heated material

6,369,381: Apparatus and method for calibration of nuclear gauges

6,310,936: Thin layer nuclear density gauge

6,054,323: Method and apparatus for analyzing asphalt content

Other Publications and Manuscripts:

G.P. Rodrigue, R.E. Troxler, and J. Smart, *Microwave Measurements*, Laboratory Manual for the Georgia Institute of Technology.

R.E. Troxler and G.P. Rodrigue, *Analysis of Ferrite Loaded Rectangular Image Guide*, Prepared for The Army Ballistic Missile Defense Advanced Technology Center, Huntsville Alabama 35807, October 10, 1985.

R.E. Troxler, *High Temperature Superconducting Phase-Shifters*, A Successful Proposal to NASA's Graduate Student Researchers Program, Marshall Space Flight Center, January, 1989.

R.E. Troxler, *Magnetic Control of Superconducting Phase-Shifters with Experimental Verification*, A Doctoral Dissertation, Georgia Institute of Technology, November, 1992.

R.E. Troxler, *Field Response of Ultra-Thin Type II Superconducting Transmission Lines*, Submitted to: IEEE Transactions on Applied Superconductivity, June 1993.

R.E. Troxler, *Field and Temperature Control of Superconducting Phase-Shifters*, Submitted to: IEEE Transactions on Magnetics, June 1993.

Exhibit B

TRANSPORTATION RESEARCH

CIRCULAR

Number E-C052 July 2003

Maintenance Management 2003

Presentations from the 10th AASHTO–TRB Maintenance Management Conference

July 13–17, 2003
Duluth, Minnesota

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Exhibit B

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Developing an Asphalt Solvent Testing and Approval Program in North Carolina

A. BATTLE WHITLEY, IV

North Carolina Department of Transportation

One of the primary goals of the North Carolina Department of Transportation (NCDOT) is to protect the state's natural resources and promote environmental stewardship. As such, in 1991, NCDOT directed its field forces to use biodegradable asphalt solvents. Numerous companies have developed a variety of solvent products using citrus, pine, and/or soy extracts purported to remove asphalt from tools and equipment used in patching and paving operations. While products are marketed as environmentally friendly, safe, and effective, environmental or safety issues may still exist. Initially the Department's approval process consisted of a review of the product's Material Safety Data Sheet (MSDS) and field trials. The various formats used for MSDS, the allowable practice of listing ingredients as "proprietary" or "trade secret," and the subjective nature of the field trials made this approval process difficult to administer. Unfortunately, there is no standard test regimen that satisfies the safety and environmental concerns, as well as the efficacy claims made by vendors. Considering the potential safety and environmental impacts of purchasing, storing, and using such products, the NCDOT has developed a laboratory-testing program for asphalt solvent products. Implementing this testing program not only ensures that the Department is proactively addressing environmental and personal safety issues, it will also ensure that a cost-effective product is purchased. The development of such a program could potentially benefit other state and local highway agencies in a comparable manner.

Historically, diesel fuel has been the asphalt-cleaning agent of choice. Diesel fuel was considered very effective, inexpensive, and readily available. As society has become more environmentally conscious, it has become unacceptable to use diesel fuel as an asphalt cleaning solvent. The introduction of biodegradable solvent products made from citrus and pine terpenes in the 1980s provided a much-needed alternative to the use of diesel fuel for removing asphalt from tools and equipment. Given that a primary goal of the North Carolina Department of Transportation (NCDOT) is environmental stewardship, the Division of Highways made the use of biodegradable solvents in paving and patching operations mandatory in 1991 (*1*). Each year, NCDOT uses about 60,000 gallons (227,100 liters) of asphalt solvent per year to clean hand tools, asphalt distributors, paving machines, dump trucks, and other equipment.

Although only a few products of this type were available initially, it became necessary to develop a material specification for asphalt removers as more and more companies began providing biodegradable solvents. The original specification attempted to address environmental, safety, and performance concerns; however, there still was no definitive way to determine if a product actually met the requirements set forth in the specification. For several years, the only verification of a product's validity was a review of technical data provided by the vendor, and a field test of a product sample by maintenance personnel during routine patching operations. Over time, this simplistic method of approving solvents showed many problems ranging from Material Safety Data Sheet (MSDS) interpretation and reliability to questions of how well a product actually works. Given the overwhelming amount of product competition and

the budgetary constraints of government agencies, a comprehensive testing and evaluation procedure is needed to ensure that we are purchasing and using a safe, effective, cost-efficient product.

PROBLEMS

The NCDOT specification for asphalt solvents has four primary components: (1) the product shall be biodegradable; (2) the product shall not contain any chlorinated solvents, caustics, or acids; (3) it shall have a closed-cup flash point greater than 140° F (60° C); and (4) it shall have a solvent effect on asphalt. From the start, the approval process for a biodegradable solvent required the vendor to provide an MSDS to the NCDOT, State Road Maintenance Unit for review. Many times other marketing and/or technical data would accompany the MSDS. This information would be reviewed for compliance with the material specification, and, if acceptable, a sample of the product would be provided for a road crew to use and evaluate. If the crew used the product to successfully clean bituminous residue from tools and equipment, then the product was approved for use.

As with any material purchased and used in construction or maintenance activities, an approval process is necessary to screen out substandard products. Most vendors and companies are reliable and produce quality products, but it is the responsibility of agencies to evaluate a product before accepting it for use. For example, one product marketed to NCDOT as environmentally safe was found to contain a reportable toxic substance listed in the "Special Precautions" section of the MSDS. Approving this product based only on the marketing information could have created many future problems. Even though this approval process is logical and has worked to eliminate many undesirable materials, the process is subjective and has numerous weaknesses.

The primary weakness of this approval process lies in the review of the MSDS. Material Safety Data Sheets come in many formats and are prepared by the product manufacturer. It is a widely accepted and allowable practice to list a material's ingredients as "trade secret" or "proprietary," which makes it virtually impossible to determine from the MSDS if non-desirable constituents exist. In addition, there is no single required standard for determining and reporting the flash point of a material. This leads to inconsistencies when trying to evaluate and compare the flash points of products. In addition to the varying information and formats found in MSDS, the fact that the MSDS is prepared and maintained by the material producer/vendor provides the potential opportunity for misrepresentations, which could have serious consequences.

The weaknesses of the original approval process extend beyond the MSDS. The material specification for asphalt solvents states "final acceptance shall be performance based." The evaluation of performance was very rudimentary and had no established control for comparison. Product samples were given to a maintenance patch crew to use during routine pavement maintenance operations. While this gave the product a "real world" test, the evaluation was subjective and depended upon how the product was used and the experience of the personnel using it.

Main Issues

In addition to the problems encountered with the original approval process, the issues driving the requirements in the material specification are environmental, personal safety, and performance

considerations. Industry understands the significance of these issues, realizing that agencies are pushing for natural, biodegradable, safe products to use for removing asphalt residue. (*2*)

Environmental

Aggressive chlorinated solvents were used in the past by laboratory technicians at asphalt plants in the evaluation of pavement mixes. Regulations for the storage, use, and disposal of chemicals are stricter today due to society's better understanding of environmental impacts. Past use and disposal methods have led to the contamination of former asphalt plant sites, which are currently being cleaned and monitored at great expense to the Department. When cleaning tools and equipment during maintenance operations, small amounts of asphalt solvents will unavoidably be spilled on the ground. Understanding this situation led to the reduction and elimination of the use of diesel fuel and chlorinated solvents for this purpose. The development of solvents using citrus, pine, and/or soy extracts and by-products made it possible to substitute biodegradable materials for the chlorinated and petroleum solvents.

While the initial reaction is that these biodegradable solvents will not adversely impact the environment, this has proven to be an invalid assumption. The market competition has led manufacturers to create various formulations using a variety of additives to enhance or reduce certain properties of their solvent products. While the primary ingredient in many of these solvents is a biodegradable extract, in pure form these extracts may actually be classified as a hazardous material due to a low flash point characteristic. Therefore, other components must be added to increase the flash point to an acceptable level. By the same token, the biodegradable components have different solvent abilities. In an effort to get a product to dissolve asphalt faster or more completely manufacturers and vendors may add other chemicals to the formulation. It is these additives that cause concern and dictate the need for careful evaluation of these products. This concern was validated through initial screenings of a few "environmentally friendly" marketed products used by NCDOT. Some of these products were found to have traces of toluene, benzene, and trichloroethene, which is contrary to the specification requirement that the product shall not contain chlorinated solvents, caustics, or acids.

Personal Safety, Storage, and Handling

The primary personal safety issue with the use of these materials is flash point. The U.S. Department of Transportation (USDOT) Hazardous Materials regulations define flammable liquids as having a flash point of not more than 141° F (60.5° C) (*3*). Another closely related definition is found in the Environmental Protection Agency (EPA) Hazardous Waste regulations (*4*). These regulations define an ignitable liquid as having a flash point less than 140° F (60° C). Both sets of regulations require the flash point to be determined by a closed-cup ASTM Standard. Asphalt solvents are routinely used around hot equipment such as asphalt distributors, kettles, and various patching and paving machines. Using a flammable product in such situations should be avoided.

Another personal safety issue comes directly from how the product is used in daily operations. Patch crews use asphalt solvents daily under high exposure conditions. Splash, spray, and spill exposures are a daily occurrence and as such must be accounted for in defining the material criteria. Therefore, it is a requirement in the asphalt solvent specification that the product be non-toxic and the pH of the material be essentially neutral.

Not only is flash point a significant consideration in regard to personal safety, it also is an issue for storage, handling, and transporting. Safety requirements dictate that flammables must be stored in cabinets or separate buildings designated for such materials. Most maintenance yards do not have the flammable storage capacity or the capital funds to address the separate storage needs for the quantities they typically keep on hand. In addition to storage of the material, transporting flammables requires the vehicle and containers, in specific quantities, to display an appropriate hazardous materials placard. This would necessitate employees being well versed in the USDOT regulations governing the transport of these materials, as well as require them to have additional commercial drivers license endorsements, which would create the need for additional training and re-certification programs.

Performance

The performance of a product is a key consideration when it comes to acceptance by field personnel. If a product is environmentally benign, yet does not quickly and effectively remove asphalt residue, field personnel will be reluctant to use it. Also, the time it takes a solvent product to remove asphalt directly affects the time spent cleaning equipment, which can impact the productivity of the paving or patching crew.

Determining how cost-effective a product is also requires a measure of performance. When deciding between two or more products that are environmentally and operationally safe, and equally effective at removing asphalt, price becomes an important factor. In the situation where one solvent product may perform slightly better than another, is the difference in performance significant enough to justify the difference in purchase price? The only way to answer this question is to evaluate and compare the performance of each product.

DEVELOPING THE PROGRAM

After experiencing problems with the previous approval process, and considering the potential impacts of purchasing large quantities of materials that may not meet the specification criteria, the decision was made to create a reliable, definitive testing program. Based on the three primary issues of environmental compliance, safety, and performance, a battery of tests was selected which addresses each aspect. The approval process consists of the following three tests: (1) a flash point test, (2) an environmental screening, and (3) a test that evaluates the performance of the solvent.

Flash Point Test

Both the EPA and USDOT regulations establishing the definition of flammable liquids require the flash point to be determined by using a closed cup method. Determining the flash point according to these regulations will address several problems, and will be the first test administered. The method mentioned first in both codes is ASTM D-93 (Pensky-Martens Closed Cup). Since laboratories currently contracted with NCDOT for testing other materials perform the ASTM D-93 method, this method was chosen for the flash point evaluation. Initial screenings of solvent products already used in NCDOT found that many times laboratories will only run the flash point test once at 140° F (60° C) and report the flash point as greater than or less than this temperature. For the purposes of this program, especially since a determination is

to be made as to whether or not a product may be sold to and used by NCDOT, it is necessary for the laboratory to report an exact flash point by running the test at varying temperatures. An average of three flash point results will be the final result reported and used for passing or failing the product.

Environmental Screening

The EPA 8021 method was first selected for evaluating these environmentally friendly, biodegradable solvents. For the initial screenings, vendors were required to have their product tested by an independent, certified laboratory using the 8021 method. While some vendors had their products evaluated using the prescribed method, many of the products were also screened using EPA Method 8260B. Upon further consideration of this method, it was determined that the 8260B method was more comprehensive, and potentially a more appropriate test. Before making a final determination on this method, the North Carolina Department of Environment and Natural Resources (DENR) was consulted. According to DENR, EPA Method 8260B is frequently used to determine if it is necessary to clean up spills of unknown materials. It is also an appropriate method for detecting hazardous chlorinated solvents and other volatile organic compounds (VOCs) that may be present in the products being tested. Table 1 lists the compounds that can be detected using EPA Method 8260B. Given the potentially serious impacts of spilling any of these compounds, a product will not be approved if any quantity of the listed compounds is detected in the sample tested.

Performance Test

There is no accepted standard for evaluating the performance of an asphalt solvent. However, it is logically reasonable to consider applying asphalt emulsion to a metal object, applying the solvent, and measuring the amount of asphalt removed. In fact, one vendor proposed a similar method used in their operations to evaluate the performance of their formulations on a piece of aluminum foil and bitumen residue from pruning sealant. There was still the lack of a control for comparing the performance of the solvents. Since diesel fuel had been used for years to clean asphalt from tools and equipment, it was chosen as the control solvent. Knowing that it was crucial to develop a scientifically valid test, NCDOT partnered with North Carolina State University and Dr. Akhtarhusein Tayebali, PE to develop a simple, repeatable test for determining the efficacy of an asphalt solvent. The expectation in the beginning was that diesel fuel would perform better than its biodegradable counter parts at dissolving asphalt. In order to establish a minimum performance requirement, samples of all of the asphalt solvents used by NCDOT at the time were evaluated along with diesel fuel. Surprisingly, all but one of the asphalt solvent products performed as well as, or better than diesel fuel. Figure 1 illustrates that many of these solvent products perform significantly better than diesel fuel (*5*). Based on this finding, NCDOT decided that a solvent must perform as well as diesel fuel, or better, by removing at least 16% of the asphalt sample in this test method in order to be approved for use. The test procedure is simple and can be performed easily by any physical-testing laboratory. At North Carolina State University, Dr. Tayebali is pursuing the possibility of standardizing this test method.

TABLE 1 List of Substances Detected by EPA Method 8260B

Acetone
Acetonitrile
Acrolein (Propenal)
Acrylonitrile
Allyl alcohol
Allyl chloride
Benzene
Benzyl chloride
Bis(2-chloroethyl)sulfide
Bromoacetone
Bromochloromethane
Bromodichloromethane
4-Bromofluorobenzene (surr)
Bromoform
Bromomethane
n-Butanol
2-Butanone (MEK)
t-Butyl alcohol
Carbon disulfide
Carbon tetrachloride
Chloral hydrate
Chlorobenzene
Chlorobenzene-d (IS) 5
Chlorodibromomethane
Chloroethane
2-Chloroethanol
2-Chloroethyl vinyl ether
Chloroform
Chloromethane
Chloroprene
3-Chloropropionitrile
Crotonaldehyde
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
Dibromomethane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,4-Dichlorobenzene-d (IS) 4
cis-1,4-Dichloro-2-butene
trans-1,4-Dichloro-2-butene
Dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,2-Dichloroethane-d (surr) 4
1,1-Dichloroethene
trans-1,2-Dichloroethene
1,2-Dichloropropane
1,3-Dichloro-2-propanol
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,2,3,4-Diepoxybutane
Diethyl ether
1,4-Difluorobenzene (IS)
1,4-Dioxane
Epichlorohydrin
Ethanol
Ethyl acetate
Ethylbenzene
Ethylene oxide
Ethyl methacrylate
Fluorobenzene (IS)
Hexachlorobutadiene
Hexachloroethane
2-Hexanone
2-Hydroxypropionitrile
Iodomethane
Isobutyl alcohol
Isopropylbenzene
Malononitrile
Methacrylonitrile
Methanol
Methylene chloride
Methyl methacrylate
4-Methyl-2-pentanone (MIBK)
Naphthalene
Nitrobenzene
2-Nitropropane
N-Nitroso-di-n-butylamine
Paraldehyde
Pentachloroethane
2-Pentanone
2-Picoline
1-Propanol
2-Propanol
Propargyl alcohol
$-Propiolactone
Propionitrile (ethyl cyanide)
n-Propylamine
Pyridine
Styrene
1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
Toluene-d (surr) 8
o-Toluidine
1,2,4-Trichlorobenzene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
Trichlorofluoromethane
1,2,3-Trichloropropane
Vinyl acetate
Vinyl chloride
o-Xylene
m-Xylene
p-Xylene

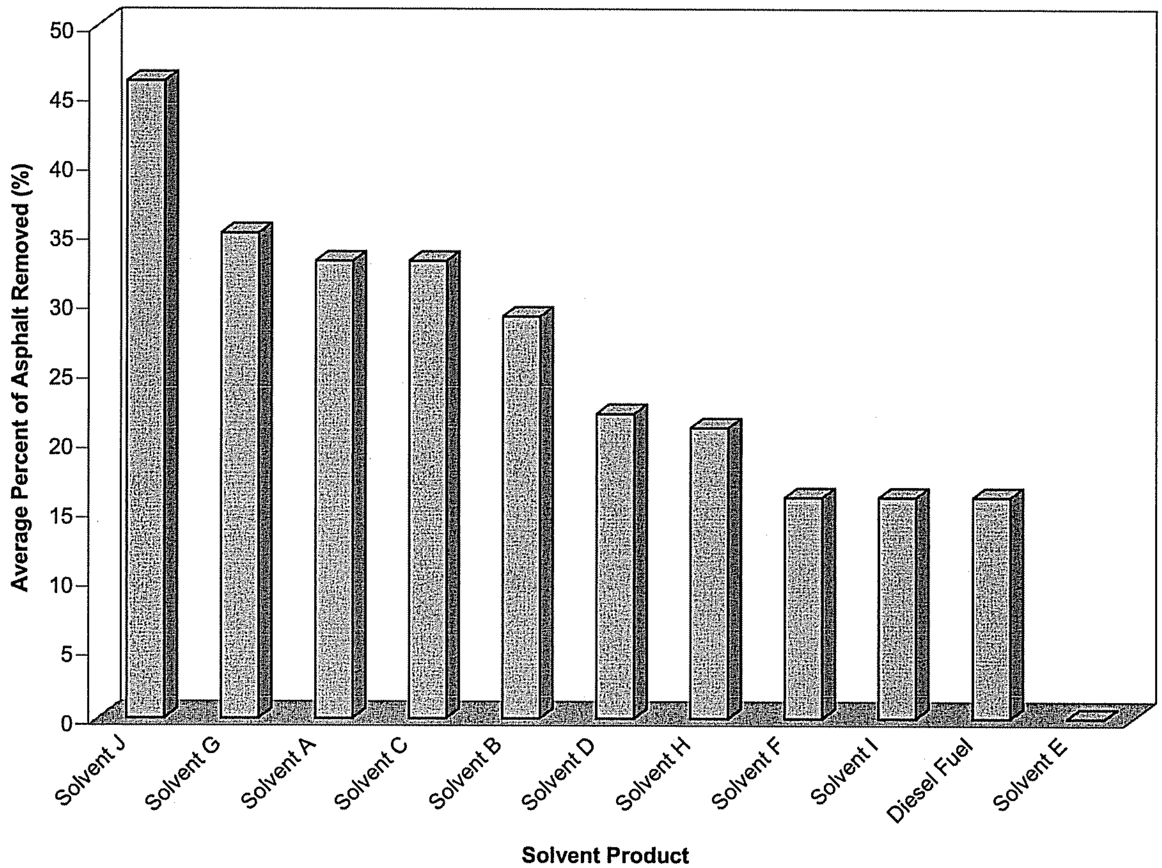

FIGURE 1 Average percentage of asphalt removed for the solvents tested.

Performance Test Procedure

The test procedure consists of applying 1.5 g of asphalt emulsion to a small aluminum dish and curing the asphalt sample at 140° F (60° C) for 24 hours. After the curing time, the dish is allowed to cool to room temperature and the weight of residual asphalt is determined. Next, 0.5g of the solvent being evaluated is applied using a dropper and allowed to work for 5 minutes. The dish is then allowed to drain upside down for 5 minutes and next rinsed thoroughly for 5 more minutes. Lastly, the dish and remaining asphalt residue is oven dried at 140° F (60° C) for 15 hours and the amount of asphalt removed is calculated.

ADMINISTERING THE PROGRAM

Now that a testing program has been defined, the approval of a solvent is handled through a multi-step process. First, any vendor seeking approval of their asphalt solvent product must submit a 2-liter (0.53-gallon) sample to the NCDOT Materials and Tests Unit (M&T). The chemical testing personnel at M&T will then split the sample and send half to a state certified laboratory for the ASTM D-93 flash point test, and the EPA Method 8260B screening. The same laboratory will test all product samples submitted for qualification. If the product passes the flash point and EPA tests, M&T will then administer the performance test using the remaining

portion of the sample. Products performing as well as, or better than, the diesel fuel control will be placed on a list of qualified asphalt solvent products. The approval process will only be performed once a year. If any additional vendors wish to have a product approved, the product would have to be submitted the next time that the approval process is opened. During the course of the year, NCDOT will randomly sample delivered asphalt solvent products and perform the flash point and EPA tests for quality assurance.

This testing and approval program has not been implemented at the time of the submission of this paper. January 2003 will be the first opportunity for vendors to submit samples for consideration, and it is expected that the resultant list of qualified products will be established by March 2003.

EXPECTED RESULTS

Given that there is a large amount of competition in this industry (*2*), it is expected that this program will increase operational efficiency by reducing the amount of time involved with the approval process. This will be done by eliminating the need to review technical information and marketing materials for products, and by limiting the time during which proposals will be allowed. Another expected benefit comes in the assurance that a safe, effective product will be purchased, without the current trial and error method many maintenance personnel now employ in deciding which product works best. Ultimately, if from this program there are sufficient qualified products available and a statewide contract for the purchase of a particular product can be established, then significant savings in the requisitioning and purchasing of asphalt solvents can be realized.

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